TRACE ELEMENTS IN SOME INDIAN COALS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY IN MINING ENGINEERING

BY

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DEPARTMENT OF MINING ENGINEERING

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CERTIFICATE

This is to certify that the thesis entitled **"TRACE ELEMENTS IN SOME INDIAN COALS"** submitted by **Sri Biswaranjan Bisoi** in partial fulfilment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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ACKNOWLEDGEMENT

I wish to express my profound gratitude and indebtedness to **Prof. D.S. Nimaje**, Department of Mining Engineering, NIT, Rourkela for introducing the present topic and for their inspiring guidance, constructive criticism and valuable suggestion throughout the project work.

I am also thankful to **Prof. B.G Mishra (HOD Chemistry Department); Prof. (Mrs.) Kakoli Karar(Paul), Department of Civil Engineering ; Prof. B.B Nayak, Department of Ceramic Engineering** for their assistance and help in carrying out different experiments in the laboratories.

Last but not least, my sincere thanks to **Mr.S.C Xess** and others who have patiently extended all sorts of help for accomplishing this undertaking.

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ABSTRACT

Better knowledge of coal quality characteristics may help to reduce some of the health problems caused by the trace elements present in coal.Trace elements can have profound adverse effects on the health of living near coal deposits ,people burning coal in homes or people burning coal in homes, living near coal deposits, coal mines, and coal-burning power plants. Trace elements such as arsenic and other hazardous elements emitted from coal-burning power plants in Europe and Asia have been shown to cause severe health problems. Perhaps the effective widespread health problems are caused by domestic coal combustion in growing countries where millions of people suffer from fluorosis and thousands from arsenic affected diseases. For example, information on concentrations and distributions of potentially toxic elements in coal may help delineate areas of a coal deposit to be avoided. Information on the modes of occurrence of these elements and the textural relations of the minerals in coal may help to predict the behaviour of the potentially toxic trace metals during coal cleaning, combustion, weathering, and leaching, so that by using modern methods of determination like AAS and AES we can find out the hazardous impacts of the trace elements present in coal.

Excess concentration of these trace elements such as cobalt, copper, selenium and zinc, and imbalances of these certain trace elements such as manganese and copper, and zinc and copper cause harmful human disease and environmental impacts. Their discoveries helped clinical scientists recognize functions of trace elements in humans and the effects of trace element deficiencies and excesses on human health. The trace elements Fe, Cu, Mn, and Zn are considered as the essential elements and their deficiency or excess may lead to several disorders in the human body. Pb and Cd are biologically nonessential and their presence in the human body above a limit is considered to be toxic and harmful. In this project the important trace elements like Arsenic (As), Iron (Fe), Nickel (Ni), Copper (Cu) and Aluminium (Al) ,which have a significant impact on environment and also on human health is discussed. To find out these trace elements in Coal experiments are being performed by using Atomic Absorption Spectroscopy.

Chapter 1

INTRODUCTION

1.1 BACKGROUND

Toxicity of coal-derived trace elements, effects of these coal-derived trace elements on water ,soil quality and effects of coal-derived radionuclides ,Recently several reports have been appeared addressing an integrated assessment of coal utilization and their environmental impacts on human health and ecosystem. Combustion of coal for the production of electrical and thermal energy is not without attendant health and environmental effects. Numerous reports have been prepared in recent years concerning these effects relative to our current pattern of coal utilization of approximately 650 million tons per year. Most studies have discussed with specific parameters of coal utilization such as health effects of coal combustion and utilization.

Trace elements and radionuclides potentially hazardous to human health and ecosystems are present in coal, which should be tested and detected for avoiding any type of harmful impacts on human and ecosystem. Trace elements of concern include, among others, As, Be, Cd, Cr, F, Hg, Ni, and Pb. Concentrations of these harmful elements vary considerably among coal types and location. Radioactive elements of concern in coal include 235U, 238U, 232Th, and associated daughter products. Concentrations of radionuclides in coal are generally less variable than those of trace elements, values of 1 ppm for U and 2 ppm for Th being reasonable national averages.

Trace and radioactive elements can enter the environment prior to coal combustion through runoff from mining, cleaning, and storage operations; during combustion in atmospheric emissions of respirable particulates and volatile elements; and following combustion through runoff and leachate infiltration into ground water from bottom ash, fly ash, and scrubber sludge deposited in settling ponds and landfills. Trace contaminants released to the environment from the coal fuel cycle may result in exposure of organisms at considerable distances due to atmospheric transport of particulate and gaseous forms, or contaminants from solid wastes (ash) may reach drinking water supplies through hydrologic transport in ground and surface waters.

Organisms, especially microorganisms in aquatic environments, can absorb, concentrate, and transform trace and radioactive elements into more concentrated forms or more toxic compounds. Biotransformation of trace and radioactive elements is particularly important in determining effects on man and other organisms, since it is the molecular form of these contaminants that determines their persistence, availability, bioaccumulation, and toxicity. Bioaccumulation is of particular concern for elements such as Cd, Hg, and Pb, because current intake levels for these substances are

near tolerable human health limits. In analytical chemistry, a trace element is an element in a sample that has an average concentration of less than 100 parts per million measured in atomic count, or less than 100 micrograms per gram. According to geochemistry, a trace element is a chemical element whose concentration is less than 1000 ppm or 0.1% of a rock's composition.

1.2 OBJECTIVE OF THE PROJECT

Here in work has been conducted to find out the trace elements that has significant impact on human health and eco-system. Some of these harmful trace elements are Nickel, Copper, Magnesium, Iron and Aluminium. So we have to conduct the experiment to find out the optimum/threshold limit of those harmful elements so that they would not have any adverse effect on the eco-system.

Chapter 2

LITERATURE REVIEW

2.1 OCCURRENCE OF TRACE ELEMENTS IN COAL

Environmental pollution by the coal based thermal power plants all over the world is cited to be one of the major sources of pollution affecting the general aesthetics of environment in terms of land usage, health hazards and air, soil and water in particular and thus leads to environmental hazards. As per the ASTM standards, in India bituminous and sub-bituminous coal results in class 'F'ash and lignite coal produces class 'C' ash, having high degree of self-hardening capacity. Physical, chemical, mineralogical, morphological and radioactive properties of these products in general vary as they are influenced by coal source/quality, combustion process, and degree of weathering, particle size and age of the coal (Adriano *et al.*, 1980; Asokan, 2000; McCarthy *et al*., 1999). One of the major concerns with coal disposal is the leaching of heavy metals to surface and underground water source, which may contaminate the ground water quality nearby the ash disposal area (Anderson *et al*., 1993; Sandhu *et al*., 1992). The trace elements in coal, such as Zn, Cd, Pb, Mo, Ni, As, Se and B are important concern for land disposal due to their environmental significance (Keefer, 1993; Spears, 2000),but the ultimate impact of each trace element will depend upon its state in Coal Combustion Residues(CCRs) and toxicity, mobility and availability in the ecosystem. The modern investigations of trace elements in coals were pioneered by (Goldschmidt in 1930), who developed the technique of quantitative chemical analysis by optical emission spectroscopy and applied it to coal ash. In these earliest works, Goldschmidt was concerned with the chemical combinations of the trace elements in coals. In addition to identifying trace elements in inorganic combinations with the minerals in coal, he postulated the presence of metal organic complexes and attributed the observed concentrations of Vanadium, Molybdenum, and Nickel to the presence of such complexes in coal.

Goldschmidt also introduced the concept of a geochemical classification of elements, in which the elements are classified on the basis of their affinities and tendencies to occur in minerals of a single group. The chalcophile elements are those which commonly form sulphides. In addition to sulphur, they include Zn, Cd, Hg, Cu, Pb, As, Sb, Se, and others. When present in coals, these elements would be expected to occur, at least in part, in sulphide minerals. Sulphides other than pyrite and

marcasite have been noted in coals, but, except in areas of local concentration, they occur in trace or minor amounts. A large numbers of silicate, sulphide, and carbonate minerals have been identified from coal seams, and the elements composing them necessarily occur in coals in inorganic combination. However, mineralogical investigations of coals have not generally been quantitative, and whether an element occurs only in inorganic combination or perhaps is also present in organic combination has not commonly been considered.

(Nicholls in 1967) approached this problem by plotting the analytical data for the concentration of a single element in coal against the ash content of the coal. Diagrams depicting a number of such points for a single coal seam or for a group of coal seams in a single geographic area were interpreted for degree of inorganic or organic affinity of the element. Nicholls concluded that
one element, boron, is entirely associated with the organic fraction in coals; some elements, such as barium, chromium, cobalt, lead, strontium, and vanadium are, in the majority of cases, associated with the inorganic fraction; and a third group including nickel, gallium, germanium, molybdenum, and copper, may be associated with either of both fractions. He then subdivided the third group into nickel and copper, which are in inorganic combination when found in large concentrations, and gallium, germanium, and molybdenum, which are largely in organic combination when found strongly concentrated.

(Horton and Aubrey in 1950) handpicked pure vitrine samples from coals and separated them into five different specific gravity fractions. They then analysed these for 16 minor elements. They concluded that for the three vitrines they studied, beryllium, germanium, vanadium, titanium, and boron were contributed almost entirely by the inherent (organically combined) mineral matter and that manganese, phosphorus, and tin were associated with the adventitious (inorganically combined) mineral matter.

The Illinois State Geological Survey has recently been extensively investigating trace elements In coal. As a part of this study four sets of float-sink samples were analysed for a number of trace and minor elements. Three coals, crushed and sized and, were separated into six specific gravity fractions by floating them in mixtures of perchloroethylene and naphtha. The heaviest of these six

fractions (1.60 sink) was then separated into two parts using bromoform (specific gravity 2.89). The fourth coal was also separated in perchloroethylene and naphtha, but only two fractions were analysed, one with specific gravity of less than 1.25 and one with specific gravity heavier than 1.60. By use of a technique similar to that of Zubovic, the trace elements determined in these samples are listed in order of decreasing affinity for the clean coal fractions, or decreasing organic affinity.

The sequences can be divided into several general groups. First, there are those elements which are always in the group most closely associated with the clean coal and which, therefore, have the greatest organic affinities. These are germanium, beryllium, and boron, which are three of the top five elements listed by Zubovic. At the other end of the list are the elements with the least affinity for the organic portion of the coal. The elements mercury, zirconium, zinc, arsenic, and cadmium are near the bottom I n all four coals studied, and lead, manganese, and molybdenum are near the bottom in three of the four. The remaining elements, those that are apparently associated to varying degrees with both the organic and inorganic portions of the coals, can also be divided into two groups: those elements that tend to be more generally allied to the elements with organic affinities (phosphorus, gallium, antimony, titanium, and vanadium) and those elements that tend to be more inorganically associated (cobalt, nickel, chromium, selenium, and copper).

The degree to which an element is associated with the lightest specific gravity fraction and therefore with the purest coal is a measure of that elements organic affinity in coal. If the element is concentrated in the heaviest specific gravity fraction, it is then in inorganic combination. Germanium, beryllium, and boron have been determined to have the greatest organic affinities, and Hg , Zr, Zn, As, Cd , Pb, Mn , and Mo are generally inorganically combined. The other elements that were determined all show degrees of inorganic and organic associations, with P, Ga,Sb, Ti , and V tending to be allied with the other elements having organic affinities and Co, N I , C r , Se, and Cu more closely associated with the inorganically combined elements. This series is very similar to a series of elements in coals presented I n order of decreasing organic affinities by Zubovic.

In general, chemical analyses of geological materials have progressed from the wet chemical methods to sophisticated instrumental methods. The major elements in the mineral constituents of

coal, Si , Al , Ti , Ca , Mg , Fe , P, S, Na, K, are the same as those in silicate rocks and are often determined by x-ray fluorescence spectroscopy and flame photometry. The minor and trace elements in coals are currently determined by several techniques, the most popular of which are optical emission and atomic absorption spectroscopy. Neutron activation analysis is also an excellent technique for determining many elements, but it requires a neutron source, usually an atomic reactor. In addition, x-ray fluorescence spectroscopy, electron spectroscopy for chemical analyses (ESCA) , and spark source mass spectroscopy have been successfully applied to the analyses of some minor and trace elements in coal.

Table 2.1 Likely modes of occurrence of 11 inorganic elements in coal and level of confidence estimate

Element	Mode of occurrence	Level of confidence
Antimony	Pyrite and accessory sulfides	4
Arsenic	Pyrite	8
Beryllium	Organic association	4
Cadmium	Sphalerite	8
Chromium	Organic or clay association	2
Cobalt	Pyrite, some in accessory sulfides	4
Lead	Galena	8
Mercury	Pyrite	6
Manganese	Carbonates, especially siderite and ankerite	8
Nickel	Unclear	2
Selenium	Organic association, pyrite and accessory sulfides, selenides	8

Table 2.2 Ranges of values for most coals (as ppm)

Element	Range	Element	Range
As	0.5–80	Mo	0.1–10
B	5–400	Ni	0.5–50
Ba	20–1000	Pb	2–80
Be	0.1–15	Sb	0.05–10
Cd	0.1–3	Se	0.2–10
Cl	50–2000	Sn	1–10
Co	0.5–30	Th	0.5–10
Cr	0.5–60	Tl	< 0.2–1
Cu	0.5–50	U	0.5–10
F	20–500	V	2–100
Hg	0.02–1	Zn	5–300
Mn	5–300		

2.2 HUMAN AND ENVIRONMENTAL IMPACTS

2.2.1 IMPACT OF ALUMINIUM

Aluminium is one of the most widely used metals and also one of the most frequently found compounds in the earth's crust. Due to these facts, aluminium is commonly known as an innocent compound. But still, when one is exposed to high concentrations, it can cause health problems. The water-soluble form of aluminium causes the harmful effects, these particles are called ions. They are usually found in a solution of aluminium in combination with other ions, for instance as aluminium chlorine.

The uptake of aluminium can take place through food, through breathing and by skin contact. Long lasting uptakes of significant concentrations of aluminium can lead to serious health effects, such as:

- Damage to the central nervous system
- Dementia
- Loss of memory
- Listlessness
- Severe trembling

Aluminium is a risk in certain working environments, such as mines, where it can be found in water. People that work in factories where aluminium is applied during production processes may endure lung problems when they breathe in aluminium dust. aluminium can cause problems for kidney patients when it enters the body during kidney dialyses.

Inhalation of finely divided aluminium and aluminium oxide powder has been reported as a cause of pulmonary fibrosis and lung damage. This effect, known as Shaver's Disease, is complicated by the presence in the inhaled air of silica and oxides of iron. May also be implicated in Alzheimer's disease. The effects of aluminium have drawn our attention, mainly due to the acidifying problems. Aluminium may accumulate in plants and cause health problems for animals that consume these plants.

The concentrations of aluminium appear to be highest in acidified lakes. In these lakes the number of fish and amphibians is declining due to reactions of aluminium ions with proteins in the gills of fish and the embryos of frogs.

High aluminium concentrations do not only cause effects upon fish, but also upon birds and other animals that consume contaminated fish and insects and upon animals that breathe in aluminium through air. The consequences for birds that consume contaminated fish are eggshell thinning and chicks with low birth-weights. The consequences for animals that breathe in aluminium through air may be lung problems, weight loss and a decline in activity.

Another negative environmental effect of aluminium is that its ions can react with phosphates, which causes phosphates to be less available to water organisms. High concentrations of aluminium may not only be found in acidified lakes and air, but also in the groundwater of acidified soils. There are strong indications that aluminium can damage the roots of trees when it is located in groundwater.

2.2.2 IMPACT OF NICKEL

Nickel is a compound that occurs in the environment only at very low levels. Humans use nickel for many different applications. The most common application of nickel is the use as an ingredient of steal and other metal products. It can be found in common metal products such as jewellery.

Foodstuffs naturally contain small amounts of nickel. Chocolate and fats are known to contain severely high quantities. Nickel uptake will boost when people eat large quantities of vegetables from polluted soils. Plants are known to accumulate nickel and as a result the nickel uptake from vegetables will be eminent. Smokers have a higher nickel uptake through their lungs. Finally, nickel can be found in detergents. Humans may be exposed to nickel by breathing air, drinking water, eating food or smoking cigarettes. Skin contact with nickel-contaminated soil or water may also result in nickel exposure. In small quantities nickel is essential, but when the uptake is too high it can be a danger to human health.

An uptake of too large quantities of nickel has the following consequences:

- Higher chances of development of lung cancer, nose cancer, larynx cancer and prostate cancer
- Sickness and dizziness after exposure to nickel gas
- Lung embolism
- Respiratory failure
- Birth defects
- Asthma and chronic bronchitis
- Allergic reactions such as skin rashes, mainly from jewellery
- Heart disorders

Nickel fumes are respiratory irritants and may cause pneumonitis. Exposure to nickel and its compounds may result in the development of a dermatitis known as “nickel itch” in sensitized individuals. The first symptom is usually itching, which occurs up to 7 days before skin eruption occurs. The primary skin eruption is erythematous, or follicular, which may be followed by skin ulceration. Nickel sensitivity, once acquired, appears to persist indefinitely.

Carcinogenicity- Nickel and certain nickel compounds have been listed by the National Toxicology Program (NTP) as being reasonably anticipated to be carcinogens. The International Agency for Research on Cancer (IARC) has listed nickel compounds within group 1 (there is sufficient evidence for carcinogenicity in humans) and nickel within group 2B (agents which are possibly carcinogenic to humans). OSHA does not regulate nickel as a carcinogen. Nickel is on the ACGIH Notice of Intended Changes as a Category A1, confirmed human carcinogen.

Nickel is released into the air by power plants and trash incinerators. It will than settle to the ground or fall down after reactions with raindrops. It usually takes a long time for nickel to be removed from air. Nickel can also end up in surface water when it is a part of wastewater streams.

The larger part of all nickel compounds that are released to the environment will adsorb to sediment or soil particles and become immobile as a result. In acidic ground however, nickel is bound to become more mobile and it will often rinse out to the groundwater. There is not much information available on the effects of nickel upon organisms other than humans. We do know that high nickel concentrations on sandy soils can clearly damage plants and high nickel concentrations in surface waters can diminish the growth rates of algae. Microorganisms can also suffer from growth decline due to the presence of nickel, but they usually develop resistance to nickel after a while.

For animals nickel is an essential foodstuff in small amounts. But nickel is not only favourable as an essential element; it can also be dangerous when the maximum tolerable amounts are exceeded. This can cause various kinds of cancer on different sites within the bodies of animals, mainly of those that live near refineries. Nickel is not known to accumulate in plants or animals. As a result nickel will not bio magnify up the food chain.

2.2.3 IMPACT OF COPPER

Copper can be found in many kinds of food, in drinking water and in air. Because of that we absorb eminent quantities of copper each day by eating, drinking and breathing. The absorption of copper is necessary, because copper is a trace element that is essential for human health. Although humans can handle proportionally large concentrations of copper, too much copper can still cause eminent health problems.

Copper concentrations in air are usually quite low, so that exposure to copper through breathing is negligible. But people that live near smelters that process copper ore into metal do experience this kind of exposure. People that live in houses that still have copper plumbing are exposed to higher levels of copper than most people, because copper is released into their drinking water through corrosion of pipes.

Occupational exposure to copper often occurs. In the work place environment copper contagion can lead to a flu-like condition known as metal fever. This condition will pass after two days and is caused by over sensitivity. Long-term exposure to copper can cause irritation of the nose, mouth and eyes and it causes headaches, stomach-aches, dizziness, vomiting and diarrhoea. Intentionally high uptakes of copper may cause liver and kidney damage and even death. Whether copper is carcinogenic has not been determined yet. There are scientific articles that indicate a link between long-term exposure to high concentrations of copper and a decline in intelligence with young adolescents. Whether this should be of concern is a topic for further investigation.

Industrial exposure to copper fumes, dusts, or mists may result in metal fume fever with atrophic changes in nasal mucous membranes. Chronic copper poisoning results in Wilson's disease, characterized by a hepatic cirrhosis, brain damage, demyelination, renal disease, and copper deposition in the cornea. When copper ends up in soil it strongly attaches to organic matter and minerals. As a result it does not travel very far after release and it hardly ever enters groundwater. In surface water copper can travel great distances, either suspended on sludge particles or as free ions.

Copper does not break down in the environment and because of that it can accumulate in plants and animals when it is found in soils. On copper-rich soils only a limited number of plants has a chance of survival. That is why there is not much plant diversity near copper-disposing factories. Due to

the effects upon plants copper is a serious threat to the productions of farmlands. Copper can seriously influence the proceedings of certain farmlands, depending upon the acidity of the soil and the presence of organic matter. Despite of this, copper-containing manures are still applied.

Copper can interrupt the activity in soils, as it negatively influences the activity of microorganisms and earthworms. The decomposition of organic matter may seriously slow down because of this.

When the soils of farmland are polluted with copper, animals will absorb concentrations that are damaging to their health. Mainly sheep suffer a great deal from copper poisoning, because the effects of copper are manifesting at fairly low concentrations.

2.2.4 IMPACT OF IRON

Iron can be found in meat, whole meal products, potatoes and vegetables. The human body absorbs iron in animal products faster than iron in plant products. Iron is an essential part of haemoglobin; the red colouring agent of the blood that transports oxygen through our bodies.

Iron may cause conjunctivitis, thyroiditis, and retinitis if it contacts and remains in the tissues. Chronic inhalation of excessive concentrations of iron oxide fumes or dusts may result in development of a benign pneumoconiosis, called siderosis, which is observable as an x-ray change. No physical impairment of lung function has been associated with siderosis. Inhalation of excessive concentrations of iron oxide may enhance the risk of lung cancer development in workers exposed to pulmonary carcinogens. LD50 (oral, rat) =30 gm/kg. (LD50: Lethal dose 50. Single dose of a substance that causes the death of 50% of an animal population from exposure to the substance by any route other than inhalation. Usually expressed as milligrams or grams of material per kilogram of animal weight (mg/kg or g/kg).)

A more common problem for humans is iron deficiency, which leads to anaemia. A man needs an average daily intake pf 7 mg of iron and a woman 11 mg; a normal diet will generally provide all that is needed. Iron (III)-O-arsenite, pentahydrate may be hazardous to the environment; special attention should be given to plants, air and water. It is strongly advised not to let the chemical enter into the environment because it persists in the environment.

2.2.5 IMPACT OF MAGNESIUM

Humans take in between 250 and 350 mg/day of magnesium and need at least 200 mg, but the body deals very effectively with this element, taking it form food when it can, and recycling what we already have when it cannot.

There is no evidence that magnesium produces systemic poisoning although persistent over-indulgence in taking magnesium supplements and medicines can lead to muscule weakness, lethargy and confusion. Effects of exposure to magnesium powder: low toxicity & not considered to be hazardous to health. Inhalation: dust may irritate mucous membranes or upper respiratory tract. Eyes: mechanical injury or particle may embed in eye. Viewing of burning magnesium powder without fire glasses may result in "Welder's flash", due to intense white flame. Skin: embedding of particle in skin. Ingestion: unlikely; however, ingestion of large amounts of magnesium powder could cause injury. Magnesium has not been tested, but it's not suspected of being carcinogenic, mutagenic or teratogenicity. Exposure to magnesium oxide fume subsequent to burning, welding or molten metal work can result in metal fume fever with the following temporary symptoms: fever, chills, nausea, vomiting & muscle pain. These usually occur 4-12 hours after exposure & last up to 48 hours. Magnesium oxide fume is a by-product of burning magnesium.

Physical dangers: Dust explosion possible if in powder or granular form, mixed with air. If dry, it can be charged electrostatically by swirling, pneumatic transport, pouring, etc.

Chemical dangers: The substance may spontaneously ignite on contact with air or moisture producing irritating or toxic fumes. Reacts violently with strong oxidants. Reacts violently with many substances causing fire and explosion hazard. Reacts with acids and water forming flammable hydrogen gas, causing fire and explosion hazard. There is very little information available on the environmental effects of magnesium oxide fume. If other mammals inhale magnesium oxide fume, they may suffer similar effects as do humans. On an environmental spectrum of 0 -3 Magnesium oxide fume registers 0.8. A score of 3 represents a very high hazard to the environment and 0 a negligible hazard. Factors that are taken into account to obtain this ranking include the extent of the material's toxic or poisonous nature and/or its lack of toxicity, and the measure of its ability to remain active in the environment and whether it accumulates in living organisms. It does not take into exposure to the substance.

Chapter 3

EXPERIMENTAL TECHNIQUES

3.1 EXPERIMENTAL TECHNIQUES

The various experimental techniques that carried out for finding concentration of trace elements in coal are:

1. Atomic Absorption Spectroscopy

2. Flame Emission Spectroscopy

3. Inductively Coupled Plasma Spectroscopy

4. Atomic Fluorescence Spectroscopy

Atomic spectra consist of **pure electronic transitions** and appear as **sharp, narrow lines,** width ~0.01 nm. All the transition intensity is confined to the sharp line, so extinction coefficients are extremely high. Compare molecules, where the transition intensity may be spread out over 100 nm. The total transition intensity is the integrated intensity over the whole band.

3.1.1 ATOMIC ABSORPTION SPECTROSCOPY (AAS):

The technique is like UV-visible absorption spectrometry with these modifications:

- A special radiation source, called a **hollow cathode lamp,** is used in place of the broad wavelength sources normal in UV-visible. Very occasionally, people have used lasers (in a research setting)

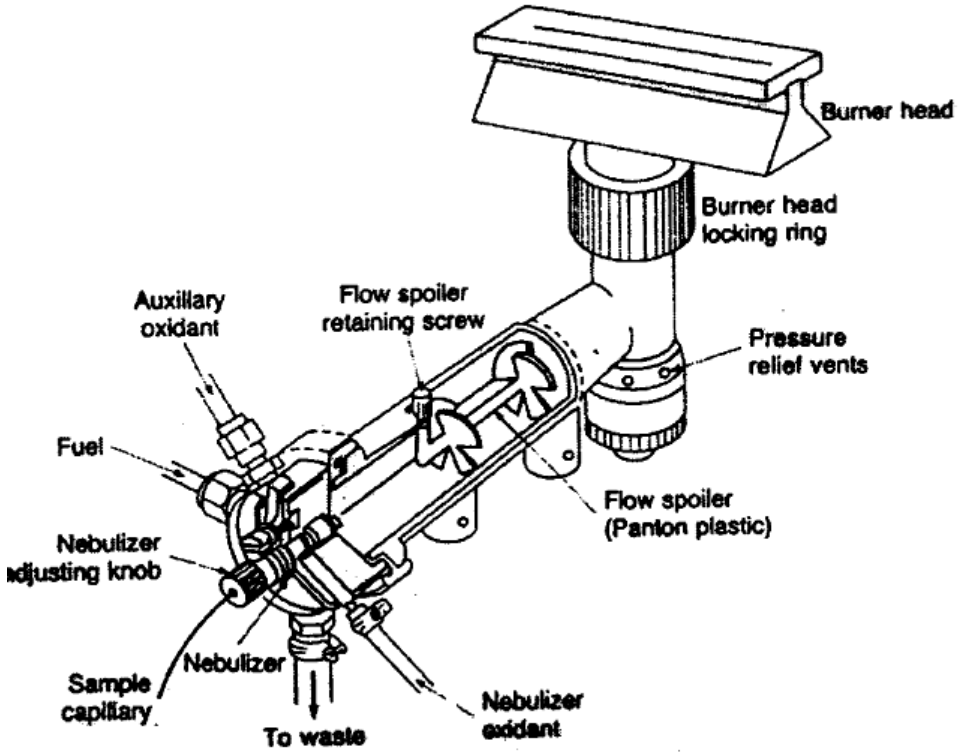
- Analyte is not quantitated directly, but is (partly) converted to gaseous atoms in a hot **flame.** Special equipment (a **nebulizer)** is used to introduce the sample into the flame.

STEPS OF EXPERIMENTS

- The analyte is prepared in aqueous solution

- The solution is aspirated into the flame, using nebulizer

Fig. 3.1



- The solvent evaporates
- the gaseous analyte decomposes, and some of it is converted to gaseous atoms
- the gaseous atoms absorb radiation from the hollow cathode lamp
- the absorbance of the sample is determined by comparing I_0 (when no analyte is present in the flame) and I_{trans}

AAS is useful because € is extremely large, and hence there is a large calibration sensitivity and low detection limit.

Approximate flame	**temperatures (K):**
Natural gas/air	2000
Hydrogen/oxygen	2900
Acetylene/oxygen	3400
Acetylene/nitrous oxide	3100

3.1.2 AAS USING GRAPHITE FURNACE (ELECTROTHERMAL AAS):

Alternative heating source to a flame. A small is deposited in a machined-out space in a cylindrical graphite block. Electrical current passed through the block heating it rapidly to >2000c.Lihgt passes through the cylindrical block, which is equipped with quartz end windows.

Advantages of Graphite furnace:

- Small shape and size makes it easier for use and better result can be acquired.
- More efficient atomization, leading to lower detection limit, down to 10^{-10} -10^{-13} grams of analyte per sample.

Disadvantages of Graphite furnace:

- Poor precision compared with flame ($\pm$ 5- 10 % compared with 1%)
- Slower: have to take the furnace apart between samples .

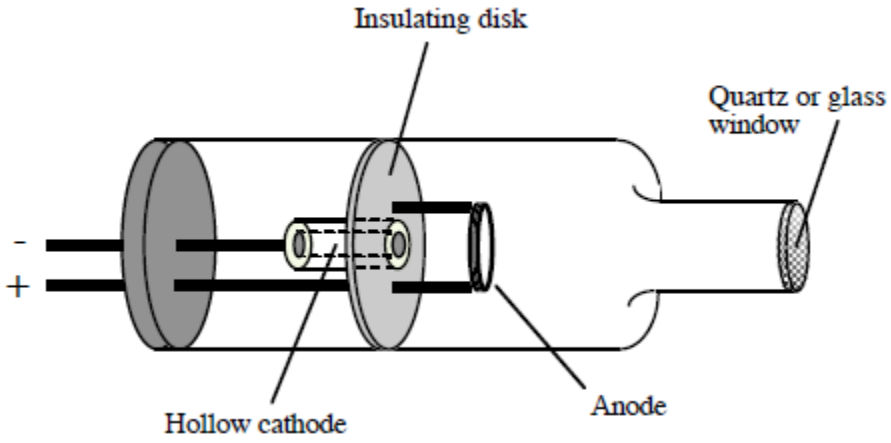


Fig. 3.2

Because atomic spectra are **lines,** the absorbing and emitting wavelengths are identical.... contrast molecular spectra, for which

$$\lambda_{emission} > \lambda_{excitation}$$

Generally, a different hollow cathode lamp is needed for each element to be studied. These are costly ($500-$ 1000 each)

The radiation emitted by hollow cathode lamp is of exactly the correct wavelength to be absorbed by the analyte. No need of monochromator analyte ahead. The monochromator in AAS spectrometer is placed between the detector and the flame in order:

1. To allow monitoring one line in the spectrum of the analyte
2. To minimise the emission from the flame itself (remember that the detector detects photon over a wide wave length range.

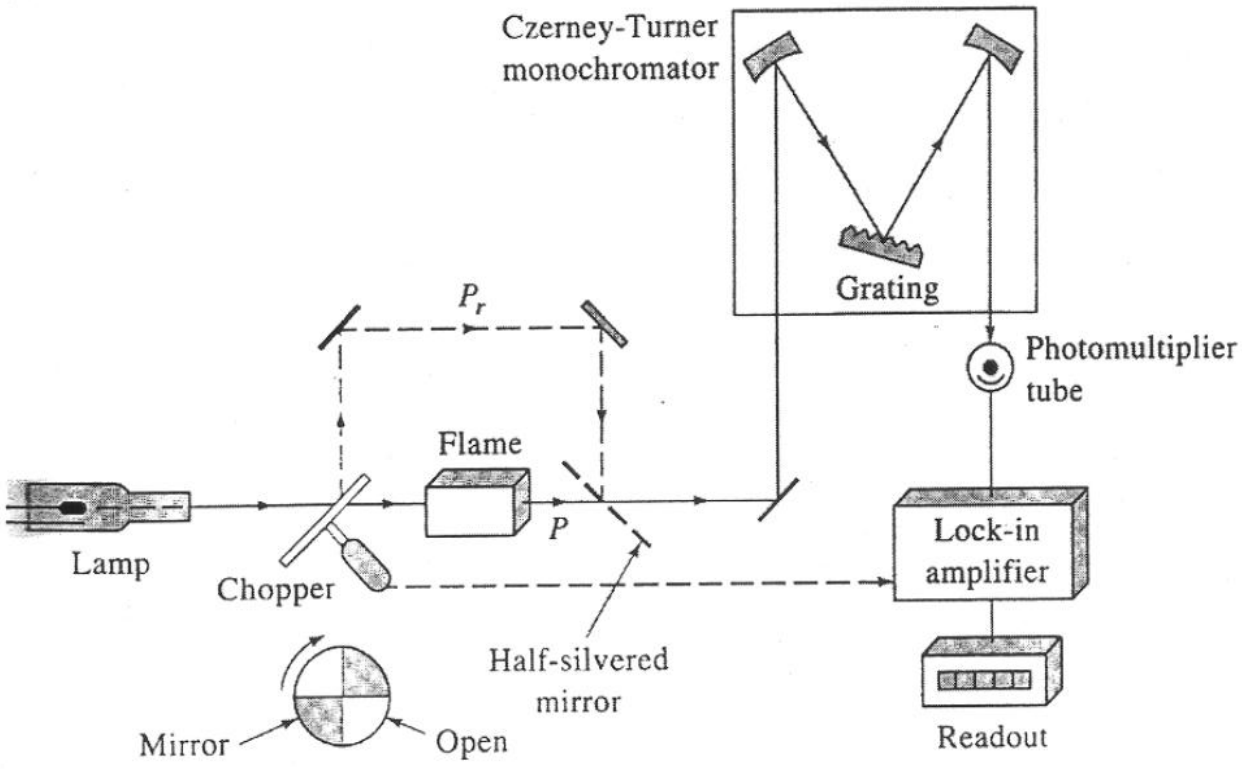


Fig. 3.3

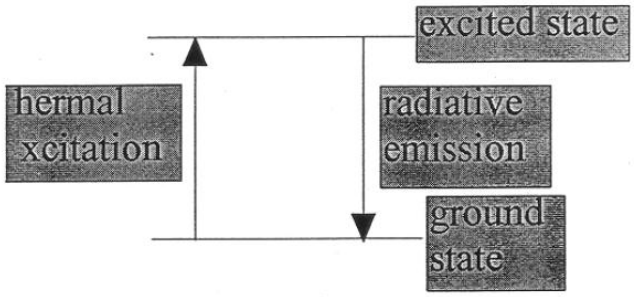
3.2 FLAME EMISSION SPECTROSCOPY

- It is of the same instrument as AAS, no flame but uses higher temperature flame in order to excite atoms more efficiently. The flame is responsible for converting the analyte to gaseous atom and then promoting some of the gas phase atom to an electronic excited state

Boltzmann equation: For N_0 atoms in the ground state and N in the excited state

$$N/N_0 = e^{-E/kT} \text{ (or } e^{-E'/RT})$$

Observe $A^* \rightarrow A + h\nu$



Element	Emission line	ΔE (eV)	N/N_0 at:	2000 K	3000 K
Cs	852	1.45		4×10^{-4}	7×10^{-3}
Na	589	2.10		1×10^{-5}	6×10^{-4}
Ca	423	2.93		1×10^{-7}	4×10^{-5}
Fe	372	3.33		2×10^{-9}	1×10^{-6}
Mg	285	4.35		3×10^{-11}	1×10^{-7}
Zn	214	5.80		7×10^{-15}	6×10^{-10}

Fig. 3.4

- The hotter flame increase the calibration sensitivity for all elements
- Resolution is FES is poorer than AAS because of pressure broadening of the atomic line width. Also the resolution is now defined by capability of the monochromator.
- The flame itself causes emission. Correct for background using dual channel detection: measure emission intensity at the emission wave length of the analyte and again a few nanometre away.

3.3 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY:

It is a method of doing multi element FES but under superior condition

- A plasma is an electrically-conducting gas which contains electrons and ions as well as neutral atoms and molecules,. Very hot flames, electric discharges and radiofrequency discharges can produce plasmas .

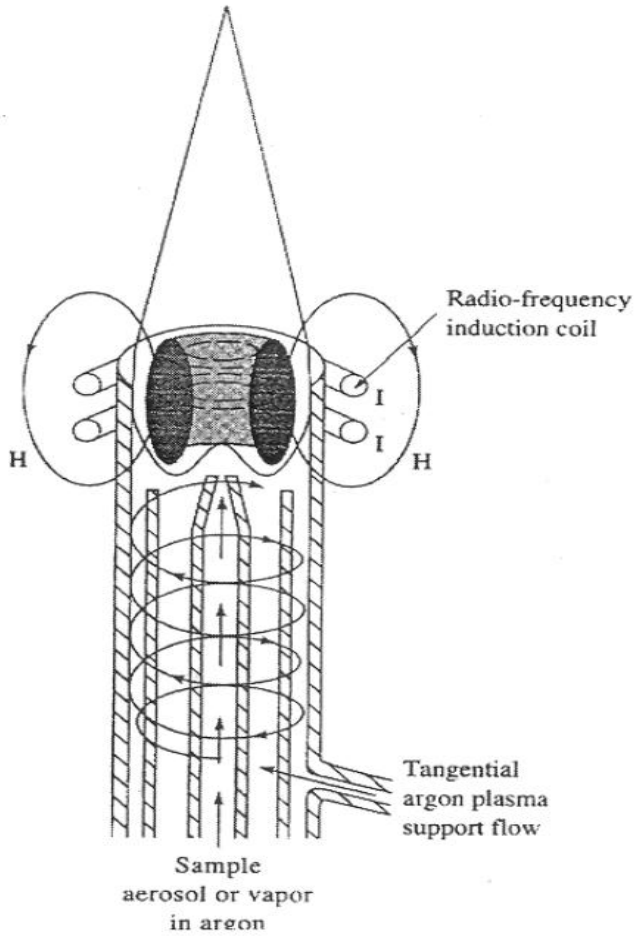


Fig. 3.5

- ICP normally uses an argon ion plasma (also called an "ion torch")which is "ignited" by RF discharge. It contains Ar(g), Ar^{+}(g) and e^{-}. It is very clean flame that has little background emission (away from characteristic lines of Ar^{*},Ar^{+*},etc)
- The flame itself cause some emission. Correct for background using dual channel detection : measure emission intensity at the emission wavelength of the analyte and again a few nm away..

3.3.1 ADVANTAGES OF ICP:

- Very high temperature ,~6000K :no problem with refractory oxides or chemical interferences ;sufficient formation of excited state analyte atoms
- Invert environment (Ar gas) prolongs life time of analyte atoms, increases steady state concentration, so detection limits are low
- Excellent dynamic range (10^{6}) makes it possible to analyse major and trace component simultaneously
- Accuracy, precision , detection limit all good for most elements

- Multi element capability :plasma conditions allow near optimal detection of most elements without changing instrument setting whether they are major or minor component of a mixture. Most elements detected at 1-10 ppm

3.4 ATOMIC FLUORESCENCE SPECTROSCOPY:

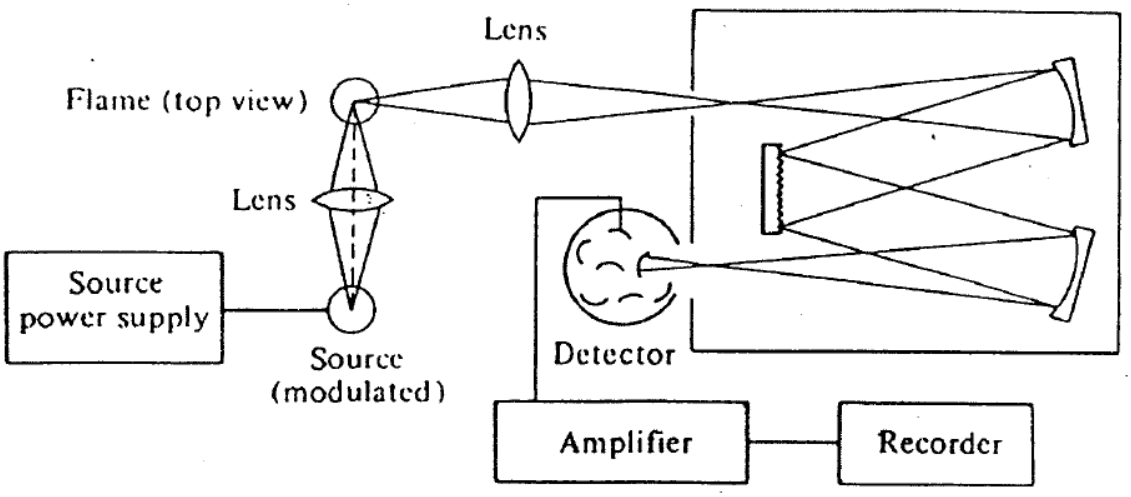


Fig. 3.6

- Source is a hollow cathode lamp
- Most observe fluorescence at 90 degree to the incident beam, or the incident beam will be seen by the detector (both have same wave length, so they are indistinguishable)
- In contrast to molecular fluorescence there is not a completely "dark background" because of emission from the flame.Also light scattering by the microscopic particles in the flame scatter some of the excitation beam into the detector .
- Source modulation to overcome the problem o emission from the flame. It works because the intensity from the hollow cathode lamp increases exponentially with temperature and therefore with rapid applied voltage.

3.5 SAMPLE COLLECTION AND PREPARATION

Sample were collected from 10 WCL mines . It is the process by which the physical and chemical properties of the mineral or ore can be ascertained with the desired accuracy. It is the process of collecting the small portion of a whole such that consistence of that portion represents that of a whole. Different types of sampling are:

- Channel sampling
- Chip sampling
- Grab sampling
- Bulk sampling
- Drill hole sampling

Chip sampling is done in hard ores where it is difficult to cut the channels. It can be taken in case of uniform ores and where the rock structures are independent of the values. The sample is collected by breaking of small equal sized chips from a face at points usually equally spaced both vertically and horizontally. Grab sampling is applied to the broken ore in the stope or at the face, ore transported. Usually grab sampling of the ore broken in the stope is unreliable as accurate estimation of the volume of broken ore is impossible. Grab sampling of tubs or ships is however more representations since samples are collected from units of regular volume. Bulk sampling is done where conventional sampling methods do not give a representative scale; large scale sampling or bulk sampling resorted to. Bulk samples eliminate the effect of irregular distribution of value or minor. For the project work, the samples have been collected by channel sampling which the most common method is followed throughout.

3.5.1 CHANNEL SAMPLING

The section of seam to be sampled shall be exposed from the roof to the floor. The seam sample shall, be taken in a channel representing the entire cross-section of the seam having the dimensions of 30 x 10 cm, that is, 30 cm in width and 10 cm in depth. For this purpose,

two parallel lines, 30 cm apart end at right angles to the bedding planes of the seam shall be marked by a chalked string on the smooth, freshly exposed surface of the seam. Obvious dirt bands exceeding 10 cm in thickness shall be excluded. The channel between the marked chalk lines in the seam shall be cut to a depth of 10 cm and the coal sample collected on a clean strong cloth or tarpaulin placed immediately at the bottom so that the chances of pieces flying off during excavation of coal are minimized.

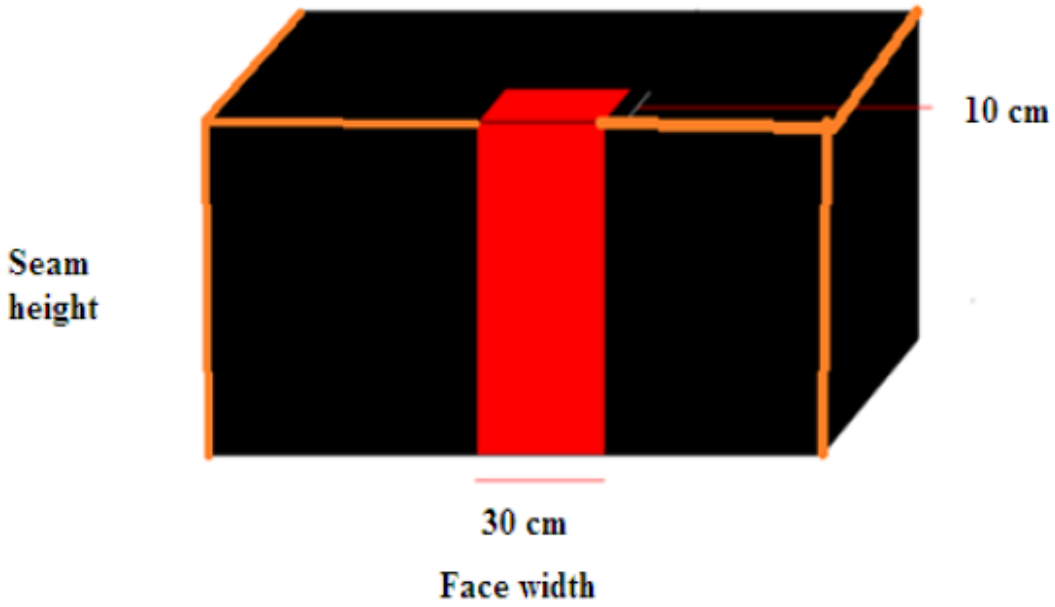


Fig. 3.7 Channel Sampling

3.5.2 SAMPLE PREPARATION

The samples received from the field via channel sampling are crushed in the laboratory as per the experimental requirements. The crushed sample is then sieved to required sizes (-250 micron) and stored in air tight polythene packets. The packets are stored in air tight containers

3.6 OPTIMISED METHOD OF COAL DIGESTION FOR TRACE METAL DETERMINATION BY ATOMIC ABSORPTION SPECTROSCOPY:

Optimised method of Coal digestion for metal determination by atomic absorption Spectroscopy:

3.6.1 STANDARDS AND REAGENTS:

Ultra high purity deionized water was used throughout the whole experimental work done on this study including: preparation of working standards, dilution of digested samples, and cleaning of digestion vessels and other equipment that was used. In addition to rinsing with ultra-pure water, digestion vessels were cleaned with trace metal grade nitric acid diluted to 10%. The multi-element standard blend contained most elements of interest at 10 mg/L, and some elements, such as Fe, Na, Mg, K, Ca, Sr, at 1000 mg/L level. Dilution was performed with the same mixture of acids and water as the one used for digestion of samples in order to imitate the matrix of analysed samples. Acids used for digestion of samples were: nitric, hydrofluoric, and hydrochloric acids. Concentration of nitric acid was 65%-70%, with boiling point of 122ºC and vapour pressure of 25 bar at 225ºC. It is an oxidizing acid that forms with all elements except: Au, Pt, Al, B, Cr, Ti, and Zr. Concentration of trace metal grade hydrofluoric acid was 47%-51%, with boiling point of 108ºC and vapour pressure of 25 bar at 240ºC. Hydrofluoric acid is a non-oxidizing acid used for decomposition of silicates. Trace metal grade hydrochloric acid had a concentration range 34%-37%, with a boiling point of 84ºC and a vapour pressure of 25 bar at 205ºC. Hydrochloric acid will form soluble chlorides with all elements except Ag, Hg, and Ti. It will also dissolve salts of weaker acids, such as carbonates, phosphates, and borates. However, it is not capable of dissolving oxides of the following elements: Al, Be, Cr, Sb, Sn, Si, Ti, and Zr.

3.6.2 EXPERIMENTAL PROCEDURES USED IN THIS STUDY:

The first part of this research involved digestion of samples using the traditional open-vessel hot-plate method published by the American Society for Testing and Materials as method number ASTM D6357-04 - “Standard Test Methods for Determination of Trace Elements in Coal, Coke, and Combustion Residues from Coal Utilization Processes by Inductively Coupled Plasma Atomic Emission Spectrometry, Inductively Coupled Plasma

Mass Spectrometry, and Graphite Furnace Atomic Absorption Spectrometry". All coal samples were prepared following the ASTM D2031- 04 method named "Standard Practice for Preparing Coal Samples for Analysis", while ash samples are used as received.

The method used for digestion of samples requires sample preconcentration by ashing. Enough coal or ash sample was measured out in order to yield approximately 0.5 g of ash that was then transferred into an open 50-mL quartz or high-silica crucible. The crucible was placed in a cold muffle furnace and temperature control adjusted so that the furnace reaches a temperature of 300°C in 1hr and then 500°C in the second hour. A temperature of 500°C was maintained for a minimum of 2 h. Ashing is considered complete when no visible evidence of carbonaceous material remains. Samples were then cooled to room temperature under conditions that minimize the absorption of water. The next step instructed by method was dissolution in acid mixture. 0.2000 to 0.5000 g of the thoroughly blended ash prepared in the previous step was placed into a 100- or 200-mL Teflon beaker. 20 mL of freshly prepared aqua regia
(3:1 mixture of hydrochloric and nitric acid) and 20 mL of concentrated hydrofluoric acid was added to the beaker. The beaker was placed on a hot plate that had been adjusted to 130 to 150°C. The mixture was heated to dryness. After the solution has evaporated, the beaker walls were rinsed with deionized water and heated again to dryness. The beaker was removed from the hot plate and cooled to room temperature. 1 mL of concentrated nitric acid and 20 mL of ultra-pure water was added and heating was resumed on a hot plate at 90 to 100°C until the sample was in the solution. The method also advises that if some residue remains after 1 h of heating, it may be ignored; trace elements are considered to be quantitatively extracted at that point. Beakers were then removed from the hot plate and the solution was allowed to cool down to room temperature. Cool solution was then filtered into 100-mL volumetric flask and diluted to volume with ultra-pure water. The solution was then transferred to a HDPE bottle to avoid adsorption of lead during storage.

The second part of this research involved digestion of the same samples used in
the previous part, but this time closed-vessel microwave digestion system was employed instead of hot-plate. Since there are no published ASTM or EPA methods for microwave assisted digestion of coal and fly ash samples, as a starting point we chose two different procedures published by Milestone in their "Application Notes" handbook that came with the

instrument. One method was used for digestion of coal samples and the other for fly ash digestion.

The method for coal digestion was a two-step process that called for a digestion of approximately 0.2g of sample in 8 mL of nitric acid with temperature ramp to 180ºC in first 5 min. and hold at 180ºC for another 10 min. Samples were then taken out of the microwave and cooled to room temperature. 2 mL of hydrofluoric acid was added and n samples were digested again with ramp to 220ºC in first 5 min. and hold at 220ºC for another 10 min. After that, samples were allowed to cool down, transferred to 50 mL plastic vials with screw caps, and diluted to the 50 mL mark.

3.7 ATOMIC ABSORPTION SPECTROPHOTOMETER (AAS)

When using atomic absorption spectrophotometry (AAS) as an analytical technique the absorption of light of free atoms is measured. Therefore it is one of the branches of atomic spectroscopy, together with flame photometry, Standard base techniques: "Flame Photometry" that measures the intensity of light emitted by free atoms when their electrons return to ground state after the excitation by light). However - unlike flame photometry - AAS is based on the "first half" of the excitation process, while atoms absorb light getting their electrons from the ground state to a higher energy level.

Although the atomic absorption spectrophotometer is quite expensive, the technique is very wide-spread, thanks to the fact that by AAS it is possible to determine about 70 elements (mainly metals) at very low concentrations. The sample is atomised at a very high temperature (2500-3000 ºC) and the free atoms have line spectrum. It means that they can only absorb the energy of light at discrete energy levels according to the excitations of electrons. Excitation energies in this case are determined by the difference between the energy level of the ground state and one of the excitation states of their electrons. Only a light with a concrete wavelength belongs to each of these excitation energies and when this light is absorbed it is missing from the continuous spectra of the electromagnetic radiation: a black line appears in the absorption spectrum of the atom. There are no vibration or rotation energy levels that would widen the lines to brands in the spectrum (like it happens in the case of UV-Vis spectrophotometry, when molecules and ions are measured, see Standard base

technique: “UV-Vis Spectrophotometry”). Using AAS free atoms are “lit” by monochromatic light (called “resonance radiation” that has got a special wavelength) that belongs to one line of their spectrum and therefore it has the suitable excitation energy mentioned above. Only the examined atoms can absorb it. As a result of absorption, the intensity of light decreases, which is proportional to the number of the examined atoms being present. That makes very sensitive quantitative measurements possible. To produce the proper monochromatic light necessary for the AAS, so called “hollow cathode lamps” are used. The cathode of this sort of lamp is made of the metal under investigation (or its alloy). It means that different lamps are used for the determination of each element. It is named after the cylindrical shape of the cathode that gives direction to emerging beam, and helps re-deposit sputtered atoms back on cathode. The anode is made of tungsten and the electrodes are surrounded by noble gases. At high voltage the cathode produces electrons that speeding up in the electric filed cause the ionisation of noble gas atoms. These high-speed noble gas ions bombard the cathode and therefore sputtering occurs, dislodging atoms from the surface of cathode. These free atoms are excited by the high-speed electrons and then emit the line spectrum characteristic of the particular element that is the cathode made of AAS called a “destructive technique”, because only solutions containing the investigated element can be used. Solid samples should be accurately weighed and then dissolved, often using strong acids (e.g. in cases when soil samples contaminated with heavy metal ions are measured). However only a very small amount of sample is enough, because of the high sensitivity of the technique.

Fig. 3.8 Photograph of an atomic absorption spectrophotometer

The solvent of the solution is evaporated and all materials present in the sample are vaporised and dissociated to atoms at the very high temperature. (The process in the reality is a bit more complicated, since ions and oxides are also produced, decomposition and association reactions take place too.) The following atomisation methods are known:

- Flame atomisation
- Graphite furnace atomisation
- Mercury hydride atomisation (this is only mentioned here, but not used while doing Standard base experiments).

The source of atoms is usually flame ("flame atomisation"). Metals could be measured at ppm concentration (part per million, that is mg kg-1 or mg dm-3 in case of dilute solutions). The sensitivity could be increased when the light travels for longer in the flame. Therefore most of the burners are about 5-10 cm long. The accuracy is very good, about 1-2%. The sample solution is sprayed ("nebulized") continuously into the flame (similarly to the flame photometer).

The graphite furnace AAS (GFAAS), a more recent technique is even more sensitive than the traditional, cheaper AAS using flame. Measurements could be done at ppb level (part per billion, ppb = 10-3 ppm, that is μg kg-1 or μg dm-3 in case of dilute solutions!). The accuracy is still about 20% in this latter case. The drying, combustion, vaporisation and atomisation of sample happen in a heated graphite tube that is placed in the way of light. This "graphite furnace" is protected against oxidation by an inert gas (e.g. argon). The other parts of the atomic absorption spectrophotometer are similar to the one used in the case of UV-Vis spectrophotometer. The monochromator that selects the proper wavelength of the emitted spectra is the usual prism or optical filter. The detector is a photo-electron multiplier that produces an electric sign proportional to the intensity of emitted light. (It contains diodes /electrodes with increasing potential/ between the photocathode and anode, where multiplied electron emission is caused by the electrons bumping in them.) The electric sign is converted and appears as absorbance on the read-out.

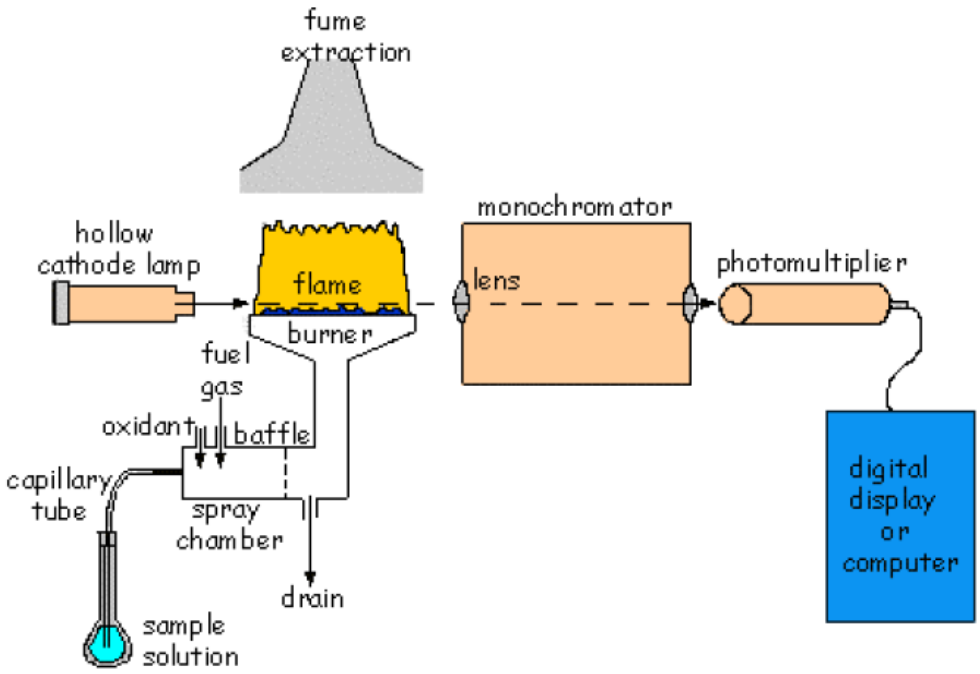


Fig. 3.9(A schematic diagram of atomic absorption spectrometer)

The evaluation of ASS measurements is also similar to the UV-Vis spectrophotometry, because it is based on Beer's Law, which says that the absorption of light is directly proportional to the number of atoms absorbing it. The more concentrated the sample solution is, the higher absorbance is measured. (For detailed explanation see Standardbase techniques: "UV-Vis Spectrophotometry". The same calibration curve or standard addition method described there could be used in case of AAS too**.**

Chapter 4

RESULTS AND DISCUSSIONS

TABLE 4.1

Concentration of Aluminium using AAS (concentration in ppm)

Sample No.	Factor	Mean (in ppm.)	Standard Deviation (S.D)	Relative S.D (in %)
1.	1.0	255.25	1.484	21.2
2	1.0	316.25	3.925	50.9
3	1.0	877.0	1.644	46.1
4	1.0	142.25	1.028	18.2
5	1.0	44.0	1.285	73.2
6	1.0	654.75	1.230	46.9
7	1.0	334.75	1.708	127.6
8	1.0	373.75	3.063	204.8
9	1.0	749.0	1.337	38.3
10	1.0	266.75	2.027	19.7

Avg. concentration / Mean in ppm.

Relative Standard Deviation in (%).

TABLE 4.2

Concentration of Copper using AAS (concentration in ppm)

Sample No.	Factor	Mean (in ppm.)	Standard Deviation (S.D)	Relative S.D (in %)
1.	1.0	5.0	0.0076	37.4
2	1.0	2.75	0.0060	56.4
3	1.0	35.5	0.0015	2.5
4	1.0	14.0	0.0030	5.3
5	1.0	13.5	0.0013	2.4
6	1.0	11.5	0.0025	5.4
7	1.0	14.25	0.0029	5.1
8	1.0	10.0	0.0021	5.3
9	1.0	9.5	0.0017	5.1
10	1.0	15.0	0.0058	2.9

Avg. concentration / Mean in ppm.

Relative Standard Deviation in (%).

TABLE 4.3

Concentration of Iron using AAS (concentration in ppm)

Sample No.	Factor	Mean (in ppm)	Standard Deviation (S.D)	Relative S.D (in %)
1.	1.0	394.0	0.0131	0.8
2	1.0	545.5	0.0434	2.0
3	1.0	176.75	0.0343	5.6
4	1.0	87.5	0.0591	4.4
5	1.0	721.0	0.0280	3.6
6	1.0	194.0	0.1662	5.2
7	1.0	355.75	0.0443	3.1
8	1.0	266.0	0.0690	6.5
9	1.0	501.0	0.0689	3.4
10	1.0	142.25	0.0459	2.9

Avg. concentration / Mean in ppm.

Relative Standard Deviation in (%).

TABLE 4.4

Concentration of Magnesium using AAS (concentration in ppm.)

Sample No.	Factor	Mean (in ppm)	Standard Deviation (S.D)	Relative S.D (in %)
1.	1.0	60.75	0.0018	0.8
2	1.0	104.5	0.0049	1.2
3	1.0	59.75	0.0046	1.9
4	1.0	63.0	0.0033	1.3
5	1.0	56.5	0.0024	1.0
6	1.0	138.0	0.0089	1.6
7	1.0	62.0	0.0025	1.0
8	1.0	49.75	0.0009	0.5
9	1.0	99.75	0.0025	6.5
10	1.0	54.75	0.0261	1.7

Avg. concentration / Mean in ppm.

Relative Standard Deviation in (%).

TABLE 4.5

Concentration of Nickel using AAS experiment (concentration in ppm.)

Sample No.	Factor	Mean (in ppm.)	Standard Deviation (S.D)	Relative S.D (in %)
1.	1.0	12.25	0.0164	33.2
2	1.0	14.5	0.0148	25.4
3	1.0	12.75	0.0184	35.9
4	1.0	16.0	0.0073	11.5
5	1.0	20.25	0.0026	3.3
6	1.0	42.86	0.0062	6.3
7	1.0	22.5	0.0093	10.3
8	1.0	29.0	0.0094	7.4
9	1.0	10.5	0.0075	11.9
10	1.0	9.75	0.0090	8.7

Avg. concentration / Mean in ppm.

Relative Standard Deviation in (%).

Chapter 5

CONCLUSIONS

1. The concentration range of Nickel varies from 0.5 to 50 ppm. From table 4.5 we conclude that WCL – 6 sample having higher concentration (42.86 ppm) of Nickel and WCL -10 having lower concentration (9.75 ppm) of Nickel . So WCL- 6 may have some adverse effect on the environment and human health also.

2. The concentration range of Magnesium varies from 5 to 200 ppm. From table 4.4 we conclude that WCL – 6 sample having higher concentration (136 ppm) of Magnesium and WCL -8 having lower concentration (49.75 ppm) of Magnesium. So WCL- 6 may have some adverse effect on the environment and human health also.

3. The maximum concentration of Iron in Indian coals is found to be 800 ppm. . From table 4.3 we conclude that WCL – 5 sample having higher concentration (721 ppm) of Iron and WCL -4 having lower concentration (87.5 ppm) of Iron. So WCL- 5 may have some adverse effect on the environment and human health also.

4..The concentration range of Copper varies from 0.5 to 50 ppm From table 4.2 we conclude that WCL – 3 sample having higher concentration (35.5 ppm) of Iron and WCL -2 having lower concentration (2.75 ppm) of Iron. So WCL- 3 may have some adverse effect on the environment and human health also.

5. The concentration range of Aluminium varies from 20 to 800 ppm. From table 4.1 we conclude that WCL – 3 sample having higher concentration (877 ppm) of Nickel and WCL -5 having lower concentration (44 ppm) of Aluminium. So WCL- 3 may have some adverse effect on the environment and human health also.

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